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interaction in both azulene and naphthalene overlayers.

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TECHNICAL REPORT NO. 3

Chemisorption and Thermal Chemistry at

Azulene and Naphthalene Adsorbed on Pt(III)

by

David Dahlgren and John C. Hemminger

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Chemisorption and Thermal Chemistry of
Azulene and Naphthalene Adsorbed on Pt(111)

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Abstract

The ordering phenomena and thermal chemistry of the isomeric aromatic hydrocarbons azulene and naphthalene have been studied on a Pt(111) surface. Complete dehydrogenation with an onset of ~ 200°C is the eventual fate of these molecules adsorbed on Pt (111). Several ordered phases exist for azulene overlayers as a function of coverage. Azulene overlayers with C/Pt atom ratios in the range 1-1.3 exhibit a <u>reversible</u> transition with temperature. The orientational ordering of neighboring molecules appears to be goverened by intermolecular forces rather than the molecule-Pt interaction in both azulene and naphthalene overlayers.

I. Introduction
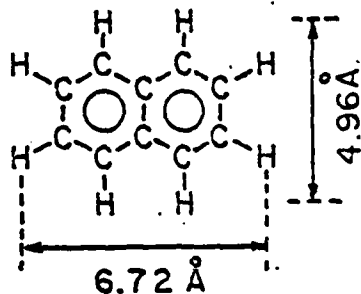
Studies of the binding and chemistry of aromatic molecules on platimum surfaces have been limited almost exclusively to benzene,[1] with some work on naphthalene.[2-4] The isomers, azulene (I) and naphthalene(II), are aromatic molecules with similar overall size and shape but with significantly different electronic structure properties.

$$
\begin{array}{cc}
\text{I} & \text{II}
\end{array}
$$

(Structure I: azulene, with dimensions 5.66 Å vertical and 7.4 Å horizontal)

(Structure II: naphthalene, with dimensions 4.96 Å vertical and 6.72 Å horizontal)
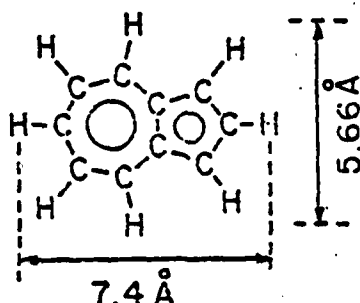
These two molecules are both essentially planar with dimensions as indicated above.[5] When the hydrogen Van der Waals radii are taken into account the linear dimensions of azulene and naphthalene are nearly identical. Azulene however has a significant dipole moment ($\mu \sim .8$ Debye[6]). Though azulene does exhibit significant aromatic stabilization energy, naphthalene is the more thermodynamically stable isomer by ~ 30 kcal/mole. The gas phase isomerization of azulene to naphthalene is complete at $400\,°C$ with an activation enthalpy of $\Delta H \sim 48$ kcal/mole.[7]

A study of the details of the binding and chemistry of azulene and naphthalene on Pt(111) should give us insight into the relative importance of structural versus electronic factors in the binding of aromatics to platinum.

II. Experimental

These experiments were carried out in an ultra high vacuum chamber (base pressure $\leq 1 \times 10^{-10}$ torr) equipped with electron optics for low energy electron diffraction (LEED) and Auger electron spectroscopy (AES) studies. The chamber is also equipped with a UTI 100C quadrupole mass spectrometer for monitoring gas exposures and for thermal desorption studies. All thermal desorption studies reported here were carried out with a linear temperature ramp. The linear temperature ramp was generated by inputing the crystal thermocouple voltage and a linear ramp into the inputs of a differential amplifier. The output of this difference amplifier was fed to the programmable heating power supply used to heat the crystal. The data acquisition from the mass spectrometer and thermocouple attached to the crystal were controlled by a microcomputer.

The azulene and naphthalene used were Aldrich reagent grade chemicals. Exposures of the crystal to azulene and naphthalene were carried out from the ambient at pressures from 5×10^{-9} torr to 5×10^{-8} torr (pressures are ion gauge readings uncorrected for sensitivity relative to N_2). The two molecules were introduced to the chamber in an identical manner. A small sample of the compound was placed in a sample tube and degassed by several freeze, pump, thaw cycles. Once degassed, the sample was warmed to 75°C and introduced to the chamber through a heated gas handling line and leak valve. The vapor pressures of azulene and naphthalene are both above 1 torr at 75°C. In the case of

azulene, care was taken to keep the gas handling line below 100°C to avoid any isomerization to naphthalene.

The Pt(111) crystal was cleaned by standard techniques of ion bombardment, annealing and oxygen treatments.[8] Care was taken to avoid any contamination of the crystal by strongly bound oxygen which has been shown to effect the chemistry of some hydrocarbons on platinum.[8,9]

III. Results

Thermal desorption experiments with azulene and naphthalene
adsorbed near room temperature on Pt(111) give very similar
results. Molecular desorption of azulene and naphthalene is not
observed following adsorption at temperatures above 0°C. The
only desorption process observed is the desorption of molecular
hydrogen. Figures 1 and 2 show the mass 2 thermal desorption
spectra following adsorption of azulene and naphthalene respec-
tively. The shapes and temperatures of the mass 2 thermal de-
sorption spectra are relatively insensitive to coverage. Fol-
lowing the desorption process the surface is covered with carbon
residue. The desorption of molecular hydrogen consists of a two
peaked spectra quite common for hydrocarbons adsorbed on pla-
tinum. The most important aspect of the desorption spectra is
that the onset of hydrogen desorption occurs at ~ 200°C. This is
well above the desorption temperature (~ 50°C) observed when
hydrogen is adsorbed on Pt(111) at room temperature.

Following room temperature adsorption at low coverage, naph-
thalene forms a disordered overlayer. At coverages nearing mono-
layer saturation (carbon to platinum atom ratio of ~ 1.1 from the
AES[10]), naphthalene forms a (6 x 3) ordered overlayer as we
have described elsewhere.[4] These naphthalene ordered over-
layers are stable at temperatures up to the onset of decom-
position at ~ 200°C. When adsorption is carried out at temp-
eratures above 0°C, saturation coverage corresponds to close
packing of naphthalene molecules with molecular centers spaced

every three platinum atoms.[4]

The ordering behavior of azulene on Pt(111) is more complex than that of naphthalene. Three distinct coverage regimes exist. At low coverage (C/Pt ratio <1.) the overlayer is disordered as in the naphthalene case at low coverage. At moderate coverage (1 $\leq$ C/Pt $\leq$ 1.3) an ordered overlayer is obtained which exhibits a LEED pattern with a _reversible_ temperature dependent transition. To our knowledge this is the first observation of a reversible ordering transition for a chemisorbed hydrocarbon. This "moderate coverage" structure exists over a very narrow range of coverages. A higher coverage phase is also observed for azulene. For C/Pt ratios greater than 1.3 a compressed coincidence structure corresponding to a (10 x 10) LEED pattern is observed. The saturation coverage for azulene on Pt(111) corresponds to a C/Pt ratio of ~ 2. .

Moderate Coverage Overlayers

The moderate coverage structures exist over a very narrow coverage range. This coverage range corresponds to C/Pt ratio from AES of 1. $\leq$ C/Pt $\leq$ 1.3. Upon adsorption resulting in this coverage range the LEED exhibits a diffuse 1/3 order ring, (Figure 3) indicative of a molecular spacing three times the Pt surface unit cell length but with domains which have a random rotational orientation with respect to the Pt substrate. If the sample is then gently annealed in vacuum at ~ 125°C an ordered overlayer is obtained. The annealed overlayer exhibits a reversible transition with temperature at ~ 130°C. At sample

temperatures below the transition temperature the LEED pattern shown in figure 4 is obtained. This consists of a (3 x 3) aligned with the Pt substrate and domains of (3 x 3) overlayers rotated 30° with respect to the Pt surface unit cell vectors. In addition some slight intensity still exists as a continuous 1/3 order ring. The background diffuse scattering is also quite high indicating some disorder. (This phase will be referred to as the α phase.) Above the transition temperature the LEED pattern shown in figure 5 is obtained (β phase) . This is a (3 x 3) overlayer aligned with the substrate with very low background, indicating a high degree of order. The temperature of this transition (α→β) is coverage dependent. At the low end of the "moderate coverage" regime the transition temperature is ∼ 100°C. The transition temperature increases with coverage up to 160°C for coverages of C/Pt ∼ 1.3. At coverages above C/Pt ∼ 1.3 the overlayer is stable up to temperatures of ∼ 190°C. The transition from the LEED pattern of figure 4 to the LEED pattern of figure 5 is completely reversible. If any of the "moderate" coverage overlayers are heated to 190°C an irreversible transition occurs to a (3 x 3) LEED pattern with increased background. At temperatures above 200°C decomposition by loss of hydrogen is facile leaving a disordered carbon overlayer.

High Coverage Overlayers

When azulene is adsorbed on Pt(111) near saturation coverage (1.4 ≤ C/Pt ≤ 2) a high coverage ordered structure is obtained. Following adsorption to this coverage range at temperatures

between 0°C and room temperature, a very poorly ordered (10 x 10) LEED pattern is observed. Annealing of the sample at ~ 125°C results in a well ordered (10 x 10) pattern as shown in figure 6. Unlike the moderate coverage overlayers these well ordered high coverage overlayers are stable at temperatures up to 180°C. When the (10 x 10) overlayer is carefully heated to 180°C it is converted irreversibly to a temperature independent "moderate" coverage structure (similar to a structure obtained for a C/Pt ratio of ~ 1.3). This consists of (3 x 3) structures aligned with the Pt and rotated by 30° with high background. No change in the AES is seen to accompany this transformation. As was seen with the moderate coverage structures, heating this overlayer to ~ 190°C results in an irreversible transition to a (3 x 3) structure aligned with the substrate with high background. Further heating leads to decomposition and evolution of H_2.

It is possible to obtain overlayers which are at intermediate coverage between the "moderate" and "high" coverage overlayers described above. These overlayers exhibit LEED patterns which are a composit of Figures 4 and 6. These intermediate coverage overlayers have temperature dependencies identical to that of a high coverage overlayer.

IV. Discussion

The results of our thermal desorption experiments for both naphthalene and azulene indicate that when adsorbed on Pt(111) both molecules are stable with respect to loss of hydrogen at temperatures below ~ 200°C. At temperatures above 200 C however decomposition by loss of hydrogen occurs. Once hydrogen is formed on a Pt(111) surface it will desorb if the surface temperature is above 50°C. At temperatures above this, hydrogen recombination and desorption will not be limiting and any structure in the mass 2 thermal desorption may be attributed to a multistep decomposition of the hydrocarbon overlayer. Naphthalene has two distinguishable types of C-H bonds. Thus it is likely that the two peaked mass 2 thermal desorption is indicative of complete removal of first one type hydrogen followed by the second type hydrogen. Sequential removal of hydrogens in this manner has been established in the case of toluene $C_6H_5(CH_3)$ adsorbed on Pt(111). Isotopically labeled studies in this case indicate the methyl group hydrogens are removed completely before the aromatic hydrogens.[12]

At coverages near saturation, the naphthalene overlayers exhibit a relatively straightforward ordering. This consists of a molecular spacing three times the Pt surface unit cell lengths with an alternation in the rotational orientation of every other molecule along one of the unit cell vectors. This leads to the observed (6 x 3) LEED pattern.[4]

The ordering phenomena for azulene on Pt(111) are significantly more complex than that of naphthalene. At low coverage the azulene overlayer is disordered. This is the same result as obtained for naphthalene.

The diagram of Figure 7 summarizes the temperature dependence of the "moderate coverage" (1. $\leq$ C/Pt $\leq$ 1.3) azulene overlayers.

While the azulene molecule is strongly bound to the platinum surface, it appears that the orientation part of the platinum-azulene interaction is relatively weak. This leads to the initial adsorption LEED behavior described in Figure 7. The 1/3 order ring observed following initial adsorption indicates a molecular spacing three times the Pt surface unit cell length. This agrees well with the amount of carbon on the surface and the size of the azulene molecule if one assumes that the molecular plane is parallel to the surface. The fact that a continuous ring is observed indicates that many domains exist with random rotational orientation with resepect to the Pt underlayer. This is also observed for naphthalene overlayers on Pt(111). In addition this phenomena is well known with respect to graphite overlayers on many metals, where the orientational part of the adsorbate metal interaction is also very weak.

Annealing these overlayers gives the molecules sufficient mobility to find the most stable orientation with respect to the Pt. It appears that the domains aligned with the Pt unit cell and those rotated by 30° with respect to the Pt unit cell are slightly more stable than randomly oriented domains. Figure 8

shows one domain (aligned with the Pt unit cell) of a possible structure. This figure clearly indicates the close packed nature of the overlayer at this coverage if the molecular plane is parallel to the surface. The α → β transition appears to correspond to an orientational order-disorder transition of neighboring azulene molecules. The details of this model of the α → β transition will be described elsewhere.[11]

The reversibility of the α → β transition virtually rules out any chemical transformation or decomposition associated with the transition. However, the irreversible high temperature transition at T ~ 190°C most likely involves a chemical modification of the azulene. The two most likely possibilities are isomerization to naphthalene, or loss of a hydrogen. Both of these processes would lead to the observed (3 x 3) LEED pattern with high background. The LEED pattern of naphthalene adsorbed on Pt(111) contains bright beams in the positions of a (3 x 3) pattern with additional weak beams to make up the (6 x 3). In addition some of the expected beams are extinguished by glide symmetries.[4] Thus with the high background observed after the transformation at 190°C it would be impossible to identify any beams other than the brightest beams in the naphthalene LEED pattern. It is also possible that the azulene may lose a hydrogen at 190°C (this is very close to the onset of the H_2 desorption in Figure 1) resulting in a molecular fragment sigma bonded to the surface. With the information available at present we cannot determine which of these processes are responsible for the 190°C transition. If isomerization to naphthalene does occur it would be likely that

some decomposition would occur also since this temperature is quite close to the onset of the H_2 desorption.

The coverage of C/Pt ~ 1.1 corresponds to saturation of naphthalene adsorption at temperatures above $0^\circ C$. However azulene forms a higher coverage phase with a C/Pt ratio between 1.3 and 2.0. The temperature dependence of this high coverage phase is shown in Figure 9. The (10 x 10) LEED pattern observed in this coverage range is obviously a compressed coincidence lattice. The most likely explanation of this overlayer involves compressing the overlayer from a molecular spacing three times the Pt surface unit cell length to a molecular spacing 2.5 times the Pt surface unit cell length. This would lead to a (5 x 5) coincidence lattice. In addition to this compression the over-layer must exhibit a doubling of the unit cell size. Such an overlayer would result in the observed (10 x 10) pattern and would be consistent with the C/Pt ratio as determined by the Auger spectra.

Two possible origins of the doubling of the unit cell are: 1) alternate rotations of the molecules as is observed in the naphthalene case, 2) the overlayer may be buckled by the mole-cules not remaining parallel to the surface and riding up on each other. Since the Auger data indicate a much higher density than would be consistent with a model with the molecular plane paral-lel to the surface we favor the second possibility with buckled overlayer. The molecular spacing 2.5 times the Pt surface unit cell length is also consistent with qualitative aspects of the relative intensities of the LEED beams. The 1/5, 2/5, 3/5, 4/5,

... order beams (every other beam) are significantly brighter than the 1/10, 3/10, 5/10, 7/10, ... order beams. This corresponds to the coincident sublattice which would be a (5 x 5) in the absence of any buckling of the azulene overlayer.

This high coverage overlayer undergoes a transition at 180 °C to what appears to be a structure at the high coverage end of the "moderate coverage" overlayer. This is most likely caused by decomposition of some of the azulene resulting in areas of the surface with a carbon overlayer. This carbon overlayer should be able to contract since a graphite like overlayer would require less area than the azulene overlayer. The remaining azulene would be allowed to relax back to the moderate coverage structures, with the temperature dependence described in Figure 7, for temperatures of 190 °C and above.

V. Summary

At first glance the chemisorption and chemistry of azulene
and naphthalene on Pt(111) appear quite dissimilar. However, on
closer inspection they turn out to be quite similar. The loss of
hydrogen occurs at quite similar temperatures. This is not sur-
prising since the C-H bond strengths in these molecules should be
similar. This also indicates that the basic interaction with the
Pt is the same. At coverages below C/Pt ~ 1 both systems are
disordered and show no signs of ordered island formation. Thus
the intermolecular interactions at these coverages and
temperatures are predominantly repulsive.

At coverages of $1 \leq C/Pt \leq 1.3$ the basic ordering phenomena
are also similar. The molecular spacing of three times the Pt
surface unit cell distance is the same for both naphthalene and
azulene. In both cases this fits very well with a model which
has the molecular plane parallel to the surface and the intermo-
lecular spacing determined by close packing rather than details
of the molecule Pt interaction. In addition, the orientational
part of the molecule - Pt interaction appears to be quite weak,
leading to the ordered ring patterns in the LEED on initial
adsorption.

The major differences between the azulene/Pt(111) and
naphthalene/Pt(111) systems involve the details of the relative
orientation of neighboring molecules. This appears to be
governed by the details of the intermolecular interactions rather
than the molecule-Pt interaction. In the case of naphthalene,

the intermolecular orientational interaction is sufficiently strong that the molecules remain oriented at temperatures up to the dehydrogenation temperature of 200°C.[4] It appears that the orientational interactions for the azulene overlayer are significantly weaker. This leads to the most straightforward explanation of the α → β transition as an orientational order-disorder transition.[11] The weakness of the orientational part of the potential for azulene may be due to an unfavorable alignment of the molecular (non-point) dipole moment parallel to the surface.

The other major difference between azulene and naphthalene adsorbed on Pt(111) is the saturation level. The fact that the azulene is stable in a buckled overlayer at higher coverage may also be due to an unfavorable alignment of the molecular dipole parallel to the surface.

This study points out some remarkable similarities between the general bonding of azulene and naphthalene to a Pt surface and the subsequent dehydrogenation chemistry. The detailed relative orientation of neighboring molecules in the overlayers is much more sensitive to the detailed intermolecular potentials, as opposed to any orientational part of the Pt-molecule inter-action. Additional work needs to be done to ellucidate the chemical transformation involved in the irreversible transition at 190°C for azulene overlayers. We plan to further investigate the reversible transition for moderate coverage azulene overlayers in an attempt to understand the intermolecular interactions in more detail.

Figure Captions

Figure 1   Thermal desorption of H_2 following adsorption of azulene on Pt(111). Linear temperature ramp ~ 13°C/sec.

Figure 2   Thermal desorption of H_2 following adsorption of naphthalene on Pt(111). Linear temperature ramp ~ 13°C/sec.

Figure 3   LEED pattern obtained after initial room temperature adsorption of azulene on Pt(111), at "moderate coverage".

Figure 4   LEED pattern obtained from the α phase of an azulene "moderate coverage" overlayer.

Figure 5   (3 x 3) LEED pattern obtained from the β phase of an azulene "moderate coverage" overlayer.

Figure 6   (10 x 10) LEED pattern obtained from the "high coverage" azulene phase after annealing.

Figure 7   Schematic representation of the temperature dependence of a "moderate coverage" azulene overlayer on Pt(111).

Figure 8   Model of the azulene "moderate coverage" α phase,
              indicating the closest packed nature of the over-
              layer if the molecular plane is parallel to the Pt
              surface.

Figure 9   Schematic representation of the temperature depen-
              dence of a "high coverage" azulene overlayer on
              Pt(111).

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H₂ DESORPTION FROM AZULENE OVERLAYER

MASS 2 SIGNAL vs CRYSTAL TEMPERATURE (°C)

Figure 1

H₂ DESORPTION FROM NAPHTHALENE OVERLAYER

MASS 2 SIGNAL vs CRYSTAL TEMPERATURE (°C)
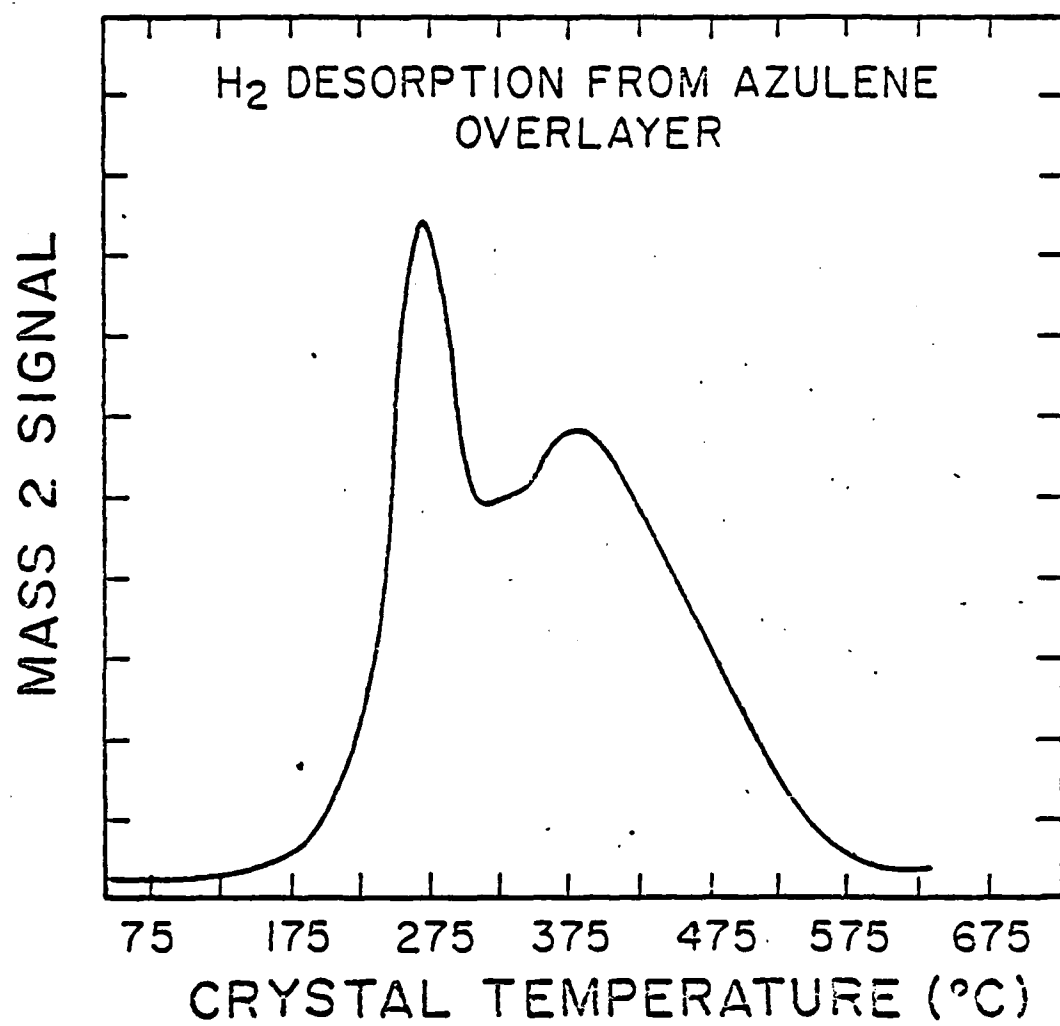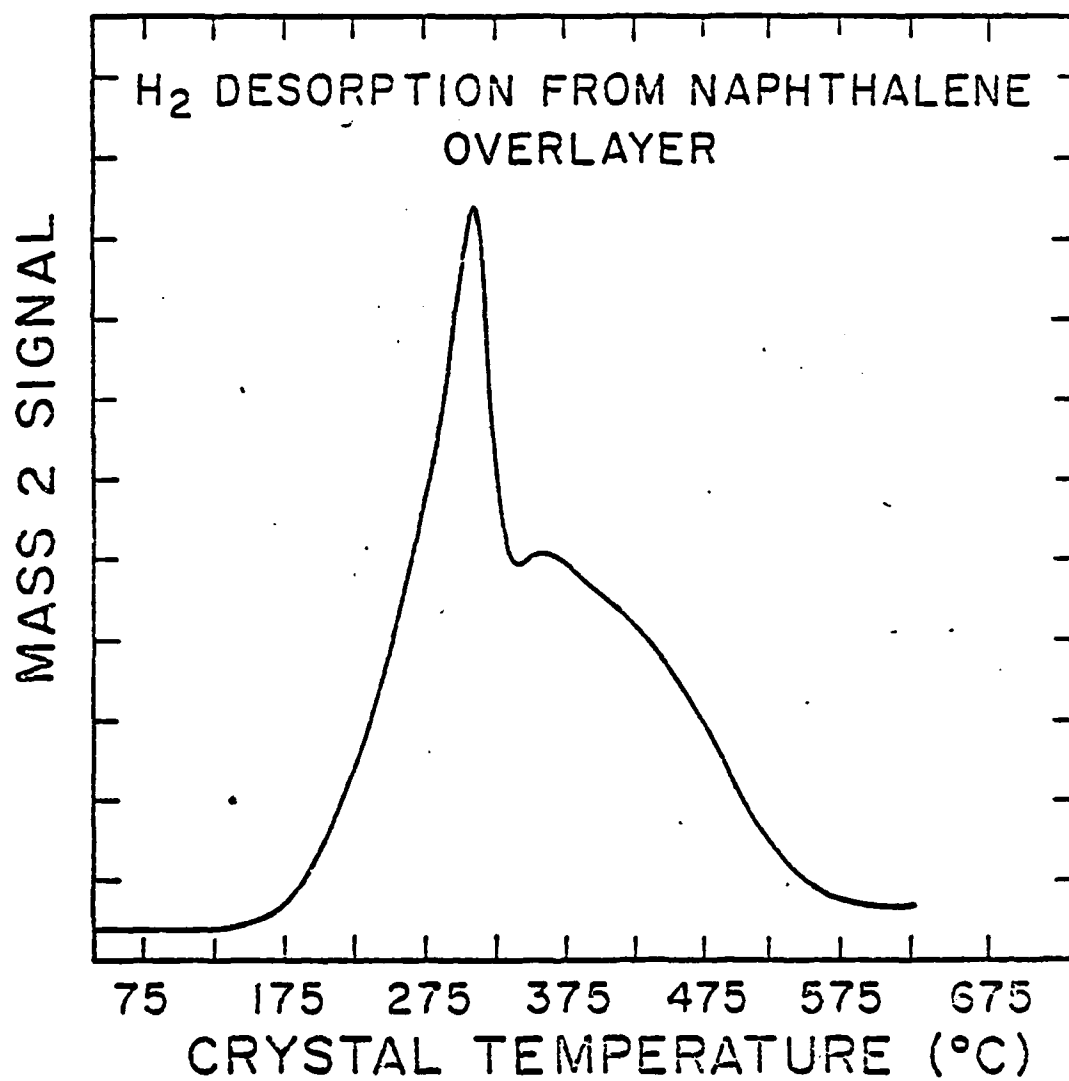
Figure 2

"Specular Beam"

"First Order
Pt Beam"
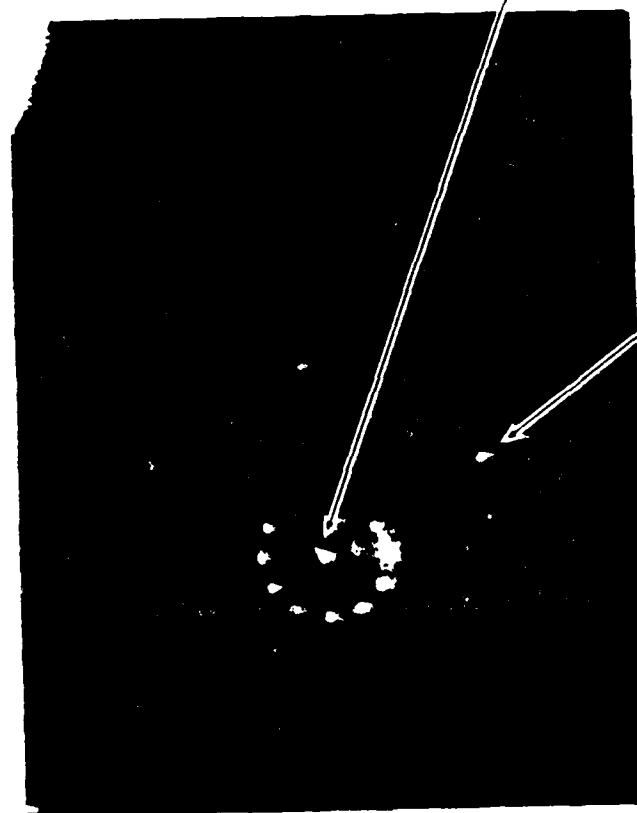
"Specular Beam"

"First Order
Pt Beam"

"Specular Beam"

"First Order
Pt Beam"

"Specular Beam"

"First Order Pt Beam"

Figure 7

TEMPERATURE DEPENDENCE OF AZULENE
"MODERATE COVERAGE" OVERLAYER

Initial Adsorption
$1 \leq c/P_1 \leq 1.3$

1/3 order ring
+
high background
(Figure 3)

Anneal
$T \sim 125°C$

(3x3)
+
(3x3) R 30°
+
high background
α phase
(Figure 4)

$T_\alpha \longrightarrow \beta$
Reversible

(3 x 3)
low background
well ordered
β phase
(Figure 5)

$T = 190°C$
Irreversible

(3 x 3)
high background
temperature
independent

$T > 200°C$
Irreversible

disordered
carbon
overlayer
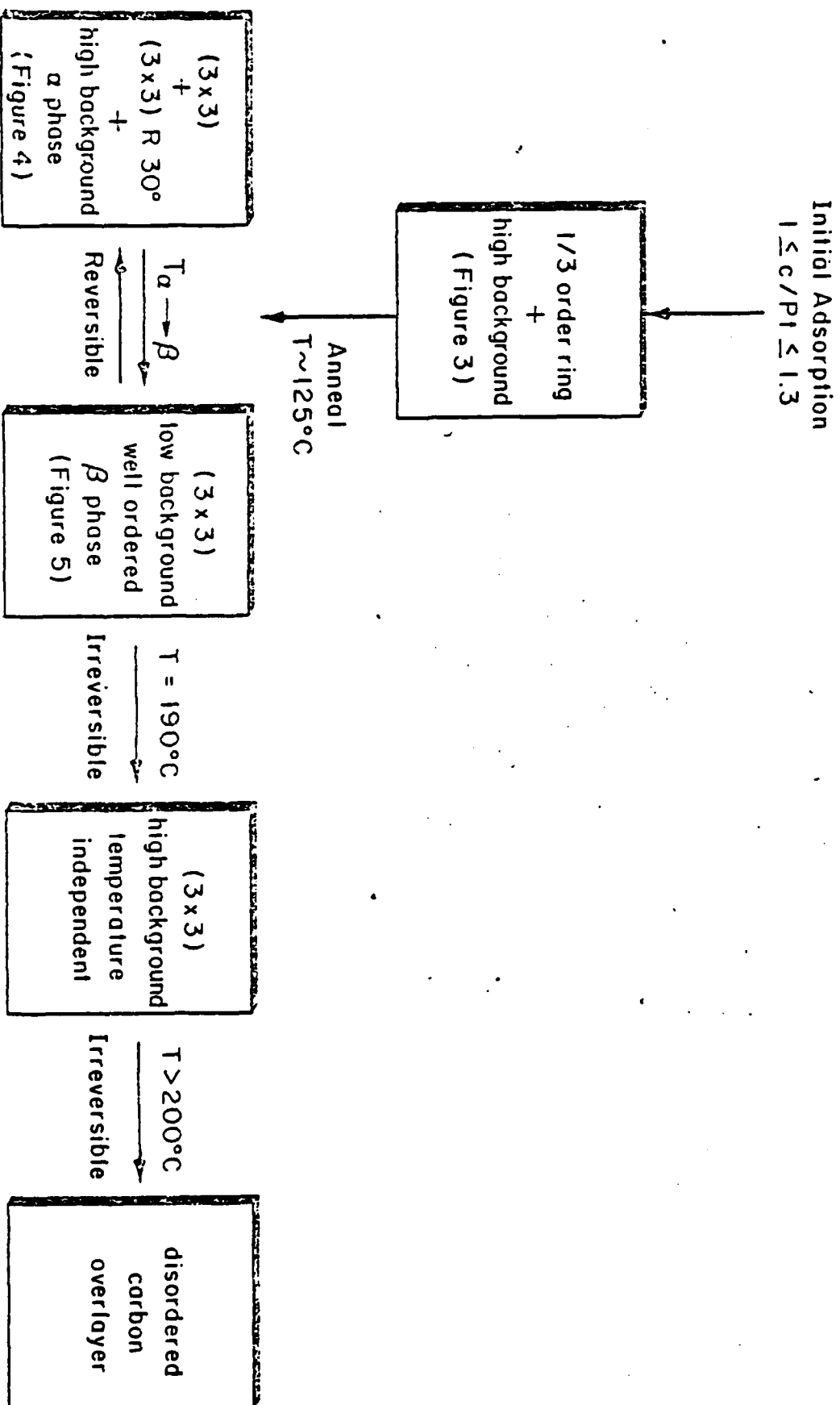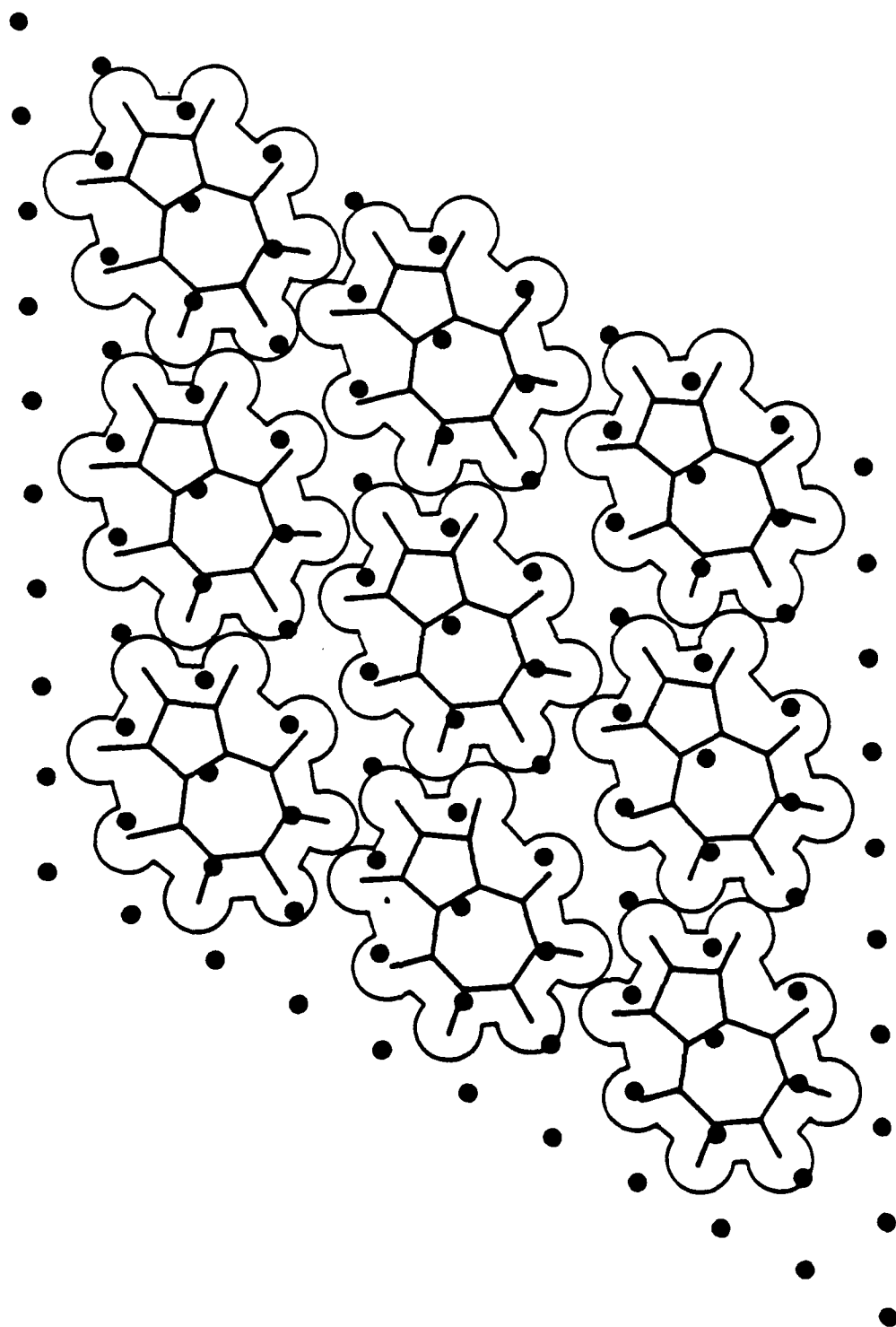
TEMPERATURE DEPENDENCE OF AZULENE
"HIGH COVERAGE" OVERLAYER

Initial Adsorption
1.3 ≤ C/PI ≤ 2

→ Highly disordered
(10 x 10)

anneal 125°C →

Well ordered
(10 x 10)
Figure 6

180°C →

(3 x 3)
+
(3 x 3) R 30°
high background
Temperature
independent below
190°C

190°C →

(3 x 3)
high background
temperature
independent

T ≥ 200°C →

disordered
carbon
overlayer

Figure 7

SP472-3/A1
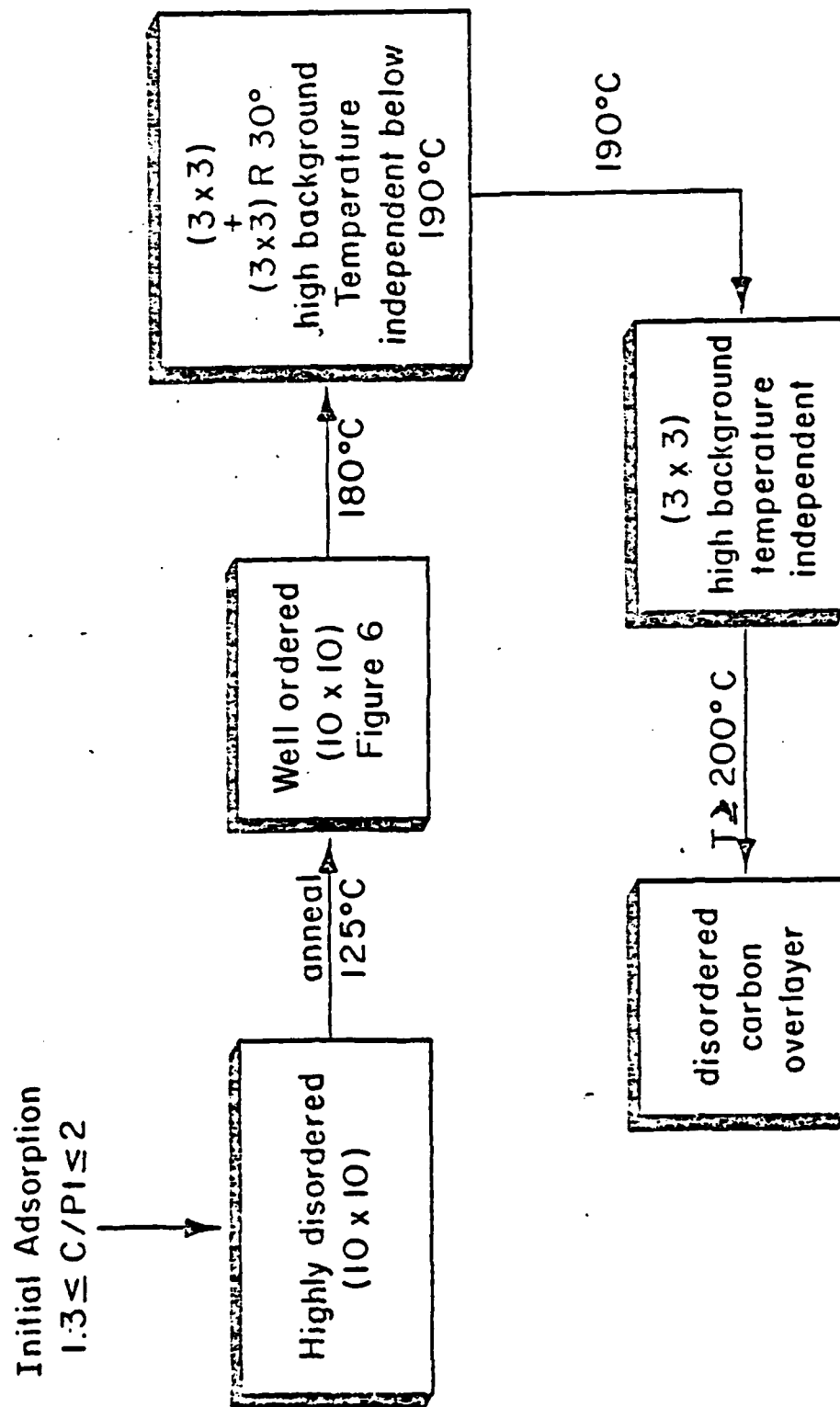
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